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STANDARD OPERATING PROCEDURE FOR VOLATILE ORGANIC COMPOUNDS (VOC) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

(GL-OA-E-038 REVISION 28)

APPLICABLE TO METHODS:

EPA SW-846 Method 8000D	EPA SW-846 Method 5030A
EPA SW-846 Method 8260B	EPA SW-846 Method 5030B
EPA SW-846 Method 8260C	EPA SW-846 Method 5035
EPA SW-846 Method 8260D	EPA SW-846 Method 5035A
Standard Methods 6200	EPA SW-846 Method 3585

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2.0 METHOD OBJECTIVES, PURPOSE, AND CODE

2.1 This procedure is used to determine purgeable organic compounds. The compounds are purged out of a solvent and absorbed onto a trap, separated via the gas chromatograph, and detected using a mass spectrometer. The Mass Selective Detector analyzes minute amounts of material by fragmenting the sample into charged ions, separating them on the basis of molecular weight-to-charge ratio, and then counting the ions as they enter the detector. The counted ions identified by mass can be plotted on a graph with the mass on the x-axis and the counted ions or abundances on the y-axis.

2.2 The procedure can be used to quantify most volatile organic compounds that:

2.2.1 Have a boiling point below 200°C.

2.2.2 Are insoluble or slightly soluble in water.

2.3 Method Codes for this procedure are EPA SW-846 Methods 8000D, 8260B, 8260C, 8260D, 5030A, 5030B, 5035A, 3585 and Standard Methods 6200.

NOTE: For South Carolina samples, only 8260B is applicable.

3.0 APPLICABLE MATRIX AND MATRICES

3.1 Applicable matrices for this method include groundwater, aqueous sludges, caustic liquids, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filtercakes, spent carbons, spent catalysts, soils, tissues, wastewaters, and sediments.

4.0 METHOD SCOPE, DETECTION LIMITS, AND PRECISION/ACCURACY MEASUREMENTS

4.1 Calibration Range: Can be as low as 0.5 ppb up to 100 ppb for most analytes. The range for those analytes with poorer purging efficiencies can be as low as 5.0 ppb up to 5000 ppb.

4.2 Tested Concentration Range: The tested concentration will be the same as the linear calibration range, with no upper limit if dilutions are performed.

4.3 Method Detection Limits (MDL) and Practical Quantitation Limit (PQL):

4.3.1 Method detection limit studies are performed annually. See GL-LB-E-001 for requirements.

4.3.2 The practical quantitation limit is defined as the lowest concentration level used to standardize the instrument.

4.4 Method precision is measured using a Statistical Process Control (SPC). The SPC limits are generated annually for surrogate, laboratory control samples, and matrix spike analyses.

4.5 Method accuracy is measured using spiked samples of known concentration.

5.0 METHOD VARIATIONS

There are no significant variations to the method procedures described in this SOP. It should be noted that method 8260C requires method-specific criteria in addition to the criteria listed

in the body of this SOP. Specific criteria are outlined in Appendix 4 and Appendix 5. Method 6200 requires method-specific criteria in addition to the criteria listed in the body of this SOP. Specific criteria are outlined in Appendix 8. For 8260D method specified criteria is outlined in Appendix 9-11.

6.0 DEFINITIONS

6.1 Calibration Standard (CAL): A solution prepared from the primary dilution, standard solution, or stock standard solutions, and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.2 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample, at the beginning of the analytical sequence. Its purpose is to determine whether the instrument calibration is in control prior to sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentration. For SC samples the CCV must come from the same source standard that is used to prepare the initial calibration curve.

6.3 Holding Times (Maximum Allowable Holding Time): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (SW-846, Table 2-40A)

6.4 Initial Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to validate an initial calibration curve with externally prepared test materials.

6.5 Initial Standard (ISTD): A known amount of standard added to a portion of the sample extract as a reference for evaluating and controlling the precision and bias of the applied analytical method.

6.6 Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, it must be a separate source standard from the initial calibration): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes of a matrix containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.7 Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank.

6.8 Limit of Quantitation (LOQ): The lowest level in the calibration curve. With the prep factor applied, the LOQ is referred to as the effective LOQ. The LOQ is equivalent to the PQL and LLOQ.

6.9 Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measured and reported. The LLOQ is the lowest point in the calibration curve and represents a concentration at which both quantitative and qualitative requirements can be consistently demonstrated. The LLOQ is verified quarterly, as the LOQ verification. The verification is performed by extracting and analyzing an LCS spiked at the lowest level of initial calibration curve (see Appendix 3 for concentrations). The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC. The LLOQ is analyzed on every instrument where data are reported and this is the laboratory's normal protocol. Recovery of target analytes in the LLOQ are compared to in-house-statistically-derived limits. Concentrations in samples reported below the LLOQ and above the MDL are qualified as estimated.

6.10 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

6.11 Method Detection Limit (MDL): The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined for analysis for a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B)

6.12 Practical Quantitation Limit (PQL): The lowest level in the calibration curve. With the prep factor applied, the PQL is referred to as the effective PQL. The PQL is equivalent to the LOQ and the LLOQ.

6.13 Quantitation Limit (DoD clarification): The value at which an instrument can accurately measure an analyte at a specific concentration (i.e., a specific numeric concentration can be quantified). These points are established by the upper and lower limits of the calibration range.

6.14 Relative Percent Difference (RPD): The difference between two duplicate samples, such as a MS/MSD, LCS/LCSD, or sample/sample dup. It is determined by taking the difference between the two results and dividing by the average.

6.15 Statistical Process Control (SPC) Limits: Statistically derived limits, which establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and surrogate standards.

6.16 Target Analytes: Identified on a list of project specific analytes of which laboratory analysis is required.

6.17 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

7.0 INTERFERENCES TO THE METHOD

7.1 Potential contaminants for this procedure include common laboratory solvents, typically ketones, methylene chloride, toluene, freons, and carbon disulfide. These compounds are used frequently in other areas of the laboratory and care must be taken to seclude the volatile organics lab and personnel from the other areas.

7.2 To minimize the contamination, use organic-free deionized water or equivalent for all analyses.

7.3 Cross contamination by carry-over can occur during sequential analyses. If cross-contamination is suspected, the affected sample or samples are reanalyzed.

7.4 The trap and other parts of the system, such as the column, are subject to contamination; therefore frequent bake out and purging of the system are required.

7.5 Sample vials are purchased as EPA Level 2 (pre-cleaned) or Level 3 (certified) and are certified free of organic contaminants. Glassware should be baked in an oven until needed. DO NOT USE HOT GLASSWARE. Wait until the glassware has cooled to room temperature before use.

8.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

8.1 Treat all chemicals and samples as potential health hazards. Exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

8.2 Personal protective equipment

8.2.1 Approved gloves are required when handling standards and samples in this procedure.

8.2.2 Work under a hood when using concentrated acids, bases, and stock solutions or if samples are suspected of having high VOC contents.

8.2.3 Safety glasses and lab coats are required when handling samples and reagents.

8.3 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling.

8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.5 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

8.6 In the event of an accident or medical emergency, seek help immediately. When time and safety permit, file an accident report form regardless of how insignificant the injury may seem.

8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

8.8 The analyst must use great care when operating and assembling instrumentation and when handling samples and reagents. Check to see that the equipment is properly assembled and connected to gas and power, etc.

8.9 In an effort to maintain instrument integrity, take care to avoid running samples that contain over-range hits of analytes, especially hydrocarbons. Observe samples for foaming. Use caution when analyzing oils, multiphasic samples or samples suspected of containing high concentrations of VOC analytes.

8.10 Refer to SOP GL-LB-N-001 the Safety, Health and Chemical Hygiene Plan for additional general safety and health information pertaining to the laboratory.

9.0 APPARATUS AND MATERIALS; REAGENTS; EQUIPMENT AND INSTRUMENTS

9.1 Apparatus and Equipment

- 9.1.1 5 mL or 10 mL glass syringes
- 9.1.2 Gas Tight syringes: 10, 25, 50, 100, 250, 500, 1000 µL
- 9.1.3 Purge vessels, 40 mL glass vials, EPA certified level 2 or higher.
- 9.1.4 Drying oven
- 9.1.5 Refrigerator and freezer
- 9.1.6 Columns
- 9.1.7 Reactor vials with mininert valves
- 9.1.8 Centrifuge
- 9.1.9 Disposable pipets
- 9.1.10 Balance
- 9.1.11 2 mL vials with screw cap lids for storage of sample extracts (soil)
- 9.1.12 Scintillation vials with screw cap lids for storage of sample aliquots (liquids)

9.2 Instrumentation

- 9.2.1 Purge and trap system for water, soil, and waste samples. This system consists of an autosampler with a heater assembly and a concentrator.
- 9.2.2 The concentrator contains a trap which holds various types of adsorbent materials. Traps are obtained from OI Analytical and are type “10”. Equivalent traps may be used from alternate vendors.
- 9.2.3 The heater assembly is used to maintain samples at 40°C.
  - 9.2.3.1 Recommended Parameters (example only)
  - 9.2.3.2 Standby: 30° C
  - 9.2.3.3 Preheat: 1.0 minute
  - 9.2.3.4 Purge: 11.0 minutes
  - 9.2.3.5 Prepurge: 0.00
  - 9.2.3.6 Sample: 40° C
  - 9.2.3.7 Dry Purge: 0.00
  - 9.2.3.8 Desorb preheat: 150° C (or the recommended temperature per manufacturer for the type of trap installed).
  - 9.2.3.9 Desorb: 0.6 minutes at 180° C
  - 9.2.3.10 Bake: 10.00 minutes at 220° C

9.2.3.11 Purge flow: 25 mL/min to 40 mL/min depending upon each instrument

9.2.3.12 Valve: 120° C, Line: 120° C, Mount: 120° C

9.3 GC/MS System

9.3.1 This device consists of a Gas Chromatograph and Mass Selective Detector, capable of scanning from 35 to 300 amu every two seconds or less. A computerized data system is used to control or assist in instrument tuning, data acquisition, data reduction, report production, and automation.

9.3.2 The laboratory utilizes Agilent 5890, 6890 and 7890 and 7890B Gas Chromatographs and 5972, 5973, 5975 and 5977A Mass Selective Detectors. Agilent Chemstation/Enviroquant Software is used for data processing and evaluations.

9.4 Columns

9.4.1 J & W DB-624 or Restek RTX-624 or equivalent fused silica capillary. column 0.25 mm ID x 60 m, 1.4 µm film thickness.

9.4.2 Temperature Limits: (-20° C to 260° C).

9.4.3 Temperature Program: used to achieve optimal analyte separation.

10.0 REAGENTS AND STANDARDS

10.1 The water used for volatile organic analysis must be high quality organic free water or equivalent.

10.2 Methanol: Spectrum high purity solvent for purge and trap analysis or equivalent. Each lot of methanol is screened for contaminants prior to use. See GL-OA-E-065 Reagent/Solvent/Standard Screening for Organic prep.

10.3 Source Standard Solutions: Most source standard solutions are purchased from approved vendors. Source standards are used to make stock, intermediate, and working standards. The primary source standards used for calibrating the instrument are listed below. The names in parentheses are an alternate description for the compound.

Calibration Short List:

Acrylonitrile	cis-1,4-Dichloro-2-butene
Acrolein	Isopropyl alcohol
Allyl chloride (3-Chloropropene)	tert-Butyl alcohol
Cyclohexanone	Isopropyl ether (Diisopropyl ether)
Ethyl methacrylate	Ethyl-tert-butyl ether (ETBE)
Isobutyl alcohol (2-Methyl-1-propanol)	Methyl-tert-amyl ether (TAME)
Methacrylonitrile	Ethyl acetate
Methyl methacrylate	Benzyl chloride
Trichlorotrifluoroethane (Freon 113)	1-Chlorohexane
Pentachloroethane	2-Nitropropane
Tetrahydrofuran (THF)	bis(2-Chloro-1-methylethyl)ether
Propionitrile (Ethyl cyanide)	1,4-Dioxane
trans-1,4-Dichloro-2-butene	2-Chloro-1,3-Butadiene (chloroprene)

Calibration Long List:

Acetone	Methyl acetate
Acetonitrile	1,2-Dichloropropane
Benzene	2,2-Dichloropropane
Bromobenzene	1,3-Dichloropropane
Bromochloromethane	cis-1,2-Dichloroethene
Bromodichloromethane	cis-1,3-Dichloropropene
Bromoform	trans-1,2-Dichloroethene
Bromomethane (Methyl Bromide)	trans-1,3-Dichloropropene
2-Butanone (Methyl Ethyl Ketone, MEK)	Ethylbenzene
Toluene-d8(surr)	Ethyl ether (Diethyl ether)
n-Butylbenzene	Hexachlorobutadiene
sec-Butylbenzene	2-Hexanone
tert-Butylbenzene	Iodomethane
Carbon disulfide	Isopropylbenzene (Cumene)
Carbon tetrachloride	p-Isopropyltoluene (p-Cymene)
Chlorobenzene	4-Methyl-2-pentanone (MIBK)
Chloroethane	Naphthalene
Chloroform	n-Propylbenzene
Chloromethane (Methyl Chloride)	n-Butyl alcohol
Cyclohexane	Styrene
Cyclohexene	Tetrachloroethylene (Perchloroethylene) (PCE)
2-Chloroethylvinyl ether	Trichlorofluoromethane (Freon II)
2-Chlorotoluene	1,1,1,2-Tetrachloroethane
4-Chlorotoluene	1,1,2,2-Tetrachloroethane
Dibromochloromethane	tert-Butyl methyl ether (MTBE)
Methylene chloride(Dichloromethane)	Toluene
1,2-Dichloroethane-d4 (surr)	Trichloroethene (TCE)
1,2-Dibromoethane (EDB) (Ethylene dibromide)	1,2,3-Trichlorobenzene
Dibromomethane	1,2,4-Trichlorobenzene
1,2-Dichlorobenzene	1,1,2-Trichloroethane
1,3-Dichlorobenzene	Methylcyclohexane
1,4-Dichlorobenzene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	1,3,5-Trimethylbenzene (Mesitylene)
1,1,1-Trichloroethane	o-Xylene
1,1-Dichloroethene	1,2-Dibromo-3-Chloropropane (DBCP)
1,1-Dichloropropene	m,p-Xylene
1,2-Dichloroethane	Vinyl acetate

Dichlorodifluoromethane (Freon 12)	Vinyl chloride
4-Bromofluorobenzene (surr)	1,2,3-Trichloropropane (TCP)

10.4 Surrogates: Toluene-d8, 4-Bromofluorobenzene and 1,2-Dichloroethane-d4. Other compounds may be used as surrogates, depending upon the analysis requirements. Final concentrations of the surrogates should be 50 µg/L. Surrogate analytes are calibrated in the Calibration Long List.

10.5 Internal standards: Fluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by the GC/MS. Final concentration of internal standards should be 50 µg/L.

10.6 BFB Tune (4-Bromofluorobenzene): Prior to sample analysis, a 5 to 50 ng BFB solution must be analyzed. The BFB solution is 50 µg/L. One microliter of this solution is placed into 5 mL of deionized water. The tune is evaluated against the criteria in Appendix 2.

10.7 Calibration Standards: A minimum of five calibration standards must be used to establish instrument linearity. Each standard is prepared in a 10 mL or 5 mL syringe. The lowest concentration level used to standardize the instrument is considered the practical quantitation limit for the compound of interest. (Refer to Appendix 3 for calibration levels)

10.8 Matrix Spiking/LCS Standards: The spiking solution is a separate source standard from the calibration standards. A set of representative compounds is used to spike sample matrices. Each compound spiked into the sample is reviewed to evaluate the effect of matrix on the spiked compounds. Refer to client specific requirements for spiking list to be reported.

10.9 Volatile organic standards are stored at -10° to -20° C.

10.10 Non-gas standards have an expiration of one month from opening. Standards for the permanent gases must be replaced after one week of opening. Intermediate standards containing gases have a one week expiration date from the day that they are made, or the vendor expiration date if sooner. Volatile working standards are made on a daily basis and are never reused the following day.

10.11 AlphaLIMS software is used to inventory and document standard traceability. For guidance on standard documentation, refer to GL-LB-E-007 for Laboratory Standards Documentation.

11.0 PRESERVATION AND SAMPLE HANDLING REQUIREMENTS

11.1 Aqueous Samples

11.1.1 Aqueous samples must be stored at 0° ≤ 6° C from the time of collection until analysis. The samples should be collected and stored with no headspace, however, pea-size bubbles are acceptable. Large volumes of sediment and any headspace should be noted in the case narrative.

NOTE: Large volumes of headspace indicate that the sample was improperly collected. Notify the Project Manager, who is responsible for contacting the client and requesting that the sample be recollected. If recollection is not possible, flag the report to indicate that the samples were improperly collected. In either case, nonconformance to proper sample collection should be noted in the case narrative.

11.1.2 Water samples should not contain any headspace and should have pH checks with pH paper strips immediately prior to analysis. Aqueous samples are preserved with hydrochloric acid to a pH less than 2 and have a 14-day holding time. Unpreserved waters have a holding time of 7 days from collection. If samples are analyzed after the 7 days with a pH above 2 the nonconformance is documented in the case narrative, the client is notified and the results are flagged accordingly. Samples that request Acrolein and Acrylonitirile must be preserved to a pH of 4-5 and analyzed with 7 days unless the applicable sampling and analysis plan or QAPP specifies otherwise.

11.1.3 Upon obtaining the aliquot for analysis, waters shall be checked for residual chlorine. The residual chlorine check is similar to the pH measurement, using a chlorine test strip. The presence of residual chlorine is documented in the runlog as "Yes" or "No". If residual chlorine is present in samples, the analyst should document the information on the runlog, in the case narrative, and notify the project manager.

11.2 Soil Samples

11.2.1 Soil samples collected for SW-846 8260 should be unpreserved or preserved in the field with specific collection kits or collected in EnCore samplers. EnCore samplers must be sent to the lab and preserved within 48 hours of collection. Preservation can be client specific however, refer to GL-OA-E-039 (SW-846 5035/SW-846 5035A, collection and preservation) for laboratory standard procedures. Method 5035 list two preservatives-sodium bisulfate and methanol. The laboratory does not analyze samples preserved in sodium bisulfate.

11.2.1.1 Low Level Soil Sample Prep

11.2.1.1.1 Using a sampling device (such as an EnCore), three sets of approximately 5 g of soil are collected per sampling site. Two are prepared for Low Level and one for High Level analysis.

11.2.1.1.2 Transfer the soil from each EnCore device to a tared 40 mL vial. Reweigh the vial (now containing the soil) and record the weight in the Soil Prep Logbook as well as on the sample label. Add 5 mL of organic free water and a magnetic stir bar before sealing the vial. Store unpreserved (water only)

samples in the freezer until analysis. Sample weights are recorded in the soil prep logbook, in AlphaLIMS in the sample prep batch and on the instrument runlog.

11.2.1.2 High Level Soils Collected in EnCore Samplers

11.2.1.2.1 Transfer the soil (approximately 5 g) from the sampling device to a tared 40 mL vial. Record the soil weight in the soil prep logbook. Add 10 mL of purge and trap grade methanol. The volume of methanol added during the extraction process can vary from 5 mL to greater than 10 mL depending on client requirements or matrix. Replace the cap and gently shake for 2 minutes. Allow the soil to settle. Centrifuge if necessary. Transfer an aliquot of the methanol extract to a 2.0 mL vial with minimum headspace, label appropriately, and store in the refrigerator until analysis. Discard the remaining soil/extract in appropriate manner for solvent/solid waste per GL-LB-G-001 for Laboratory Waste Management Plan. Sample weights are recorded in the soil prep logbook, in AlphaLIMS in the sample prep batch and on the instrument runlog.

11.2.1.2.2 A methanol solvent blank (HB) is prepared by placing a portion of the methanol used for sample extractions into a 2.0 mL vial with minimal headspace. The labeled vial is stored with the soil extracts at 0° C ≤ 6° C until the time of analysis. It is analyzed under the same conditions as the associated samples. Methanol used in the laboratory is screened by lot prior to use.

11.3 Solids or liquid waste and oils should not be acid preserved. These types of samples should be stored at 0° ≤ 6° C from the time of collection until analysis.

11.4 Analysts receive the samples directly from the storage cooler. Samples must be tracked into the analyst's custody, then tracked back to the cooler or archive upon completion of the analysis.

11.5 If samples are analyzed out of holding or the correct preservation was not performed, the analyst must immediately notify the Project Manager. Additional information for the nonconformance should be indicated in the batch and/or fractional case narrative.

12.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

12.1 Low Level Soil Sample Preservation (SW-846 5035/SW-846 5035A)

12.1.1 Preserved samples received from the field are wiped to remove any extraneous material from the exterior of the vial. The vial is weighed to the nearest 0.1 g and the weight is recorded in the soil prep logbook. The weights are entered into the AlphaLIMS system in the sample prep batch. The analyst should observe the vial for any indication for leaks or cracks. If any significant observations are detected, the project manager is notified and the appropriate action is taken.

12.1.2 Allow the sample to warm to room temperature. Shake the vial gently to ensure that the contents move freely and that stirring will be effective. Place the vial in the autosampler and ensure the autosampler will add the appropriate volumes of organic-free water, surrogates, and internal standards to all vials. All samples, blanks, and calibration standards are analyzed with the same final volume.

12.1.3 When low level soil analysis produces target analyte results with concentrations higher than the calibration range of the instrument, the sample is reanalyzed using the prepared aliquot from the High Level Soil Sample Prep (11.2.2). Such reanalysis need only address those analytes for which the concentration exceeded the calibration range of the Low Level Method. The maximum volume of methanol analyzed is 100 µL.

12.1.4 Analysts should never open a 5035 preserved low level soil vial from the field or EnCore sampler. All spiking must be performed through the vial septum with a syringe or autosampler needle.

12.1.5 Results are to be reported on a dry weight basis unless otherwise specified by the client.

NOTE: Low level soil samples originating in South Carolina must be collected and analyzed using EPA Methods 5035.

12.2 High Level Soil Sample Preparation (SW-846 5035/SW-846 5035A)

12.2.1 High Level soils collected in EnCore samplers

12.2.1.1 To analyze the methanol extract, transfer 100 µL of the extract to a 5 mL syringe containing 4.9 mL of organic-free water. Transfer the fortified water to an autosampler vial (40 mL vial with septum cap). Place the vial in the autosampler. If concentrations of analytes exceed the calibration range, the sample is reanalyzed using a smaller volume of sample aliquot.

12.2.1.2 For high level soils preserved in the field, this aliquot may or may not be analyzed depending on the concentration of the analytes present in the sample. Prior to analysis, allow the soil/methanol extract to reach room temperature. Withdraw 100 µL or the desired volume from the vial. Transfer the methanol extract to a syringe containing 4.9 mL of organic-free water.

Transfer the fortified water to an autosampler vial (40 mL volatiles vial). Place vial in autosampler.

12.2.1.3 All sample analyses for high level soils using the methanol extract procedure require the preparation and analysis of a methanol blank (HB) if the samples are prepared at the laboratory. Field prepared samples may or may not include a methanol aliquot for analysis. Place 100 µL of the methanol in a syringe containing 4.9 mL of organic-free water. Transfer the water to an autosampler vial (40 mL volatiles vial). Place the vial in the autosampler. The acceptance criteria applied to the HB should be the same as method blank criteria outlined in 17.2.3.

12.3 Low Level Soil analysis: Allow samples to reach room temperature. Weigh approximately 5 g (± 0.1 g) of sample into a 40 mL autosampler vial. Document the weight used in the soil preparation logbook and on the instrument runlog. The weights are also entered into AlphaLIMS system in the sample prep batch. Add 5 mL of organic-free water to the vial and a stir bar. Gently shake the sample vial to ensure the magnetic stir bar moves freely. Place the sample vial in the appropriate position and complete the autosampler sequence as desired to purge each sample.

NOTE: The sample may be diluted by weighing less sample. Do not analyze less than 1.0g of the solid sample. If a higher dilution is required, use the high level soil sample preparation method.

12.4 High Level Soil Preparation (SW-846 5035/SW-846 5030C)

12.4.1 Solid samples that contain concentrations of analytes greater than the highest concentration level in the calibration or samples with high viscosity, are extracted with methanol, and then diluted in water. Approximately 5 g of sample is weighed out and extracted with 10 mL of methanol. The methanol extract is gently shaken for two minutes then allowed to settle until distinct layers form. Centrifuge if necessary. An aliquot of the methanol layer is then transferred to a 2.0 mL vial. The extract is then analyzed immediately or the vial is stored in the sample refrigerator until analysis. The dilution factor is 1:50 if 100 µL of sample is used. The minimum amount of soil that can be used is 1 gram.

12.4.2 Oily waste samples or samples of unknown solid matrix are extracted following SW 846 Method 3585 with some modifications. SW 846 Method 3585 suggests n-Hexadecane or another appropriate solvent be used as the extraction solvent when performing waste dilutions on oily matrices. The laboratory uses purge and trap methanol as the extraction solvent for these types of sample preparations. The samples are prepared by weighing a 1 g aliquot into a VOA vial and adding an appropriate volume of purge and trap methanol to meet the client's detection limit requirements if possible.

This volume is usually 5 to 10 mL but can be as high as 20 mL. The methanol extract is gently shaken for two minutes then allowed to settle until distinct layers form. Centrifuge if necessary. An aliquot of the methanol layer is then transferred to a 2.0 mL vial. The methanol volume may be increased if the matrix absorbs the initial volume of methanol used. The total volume of methanol used is documented in the soil prep logbook and AlphaLIMS. Care should be taken with any oily or unknown matrix to avoid contamination to the analytical system from high concentrations of targets or non-target analytes. Methanol is screened by lot prior to use.

12.5 Aqueous analysis (5030B)

12.5.1 Allow samples to reach room temperature. Invert each sample vial to check for headspace. Gently pour approximately 6 mL of the aqueous sample into a 10 mL syringe. Place the plunger in the syringe and invert, allowing any bubbles to escape through the end fitting. Adjust the sample volume 5.0 mL by depressing the plunger to the mark. The extraneous sample volume may be added back to the original sample vial. Transfer the 5.0 mL of sample to a clean 40 mL autosampler vial and place the vial in the appropriate autosampler position. Complete the autosampler sequence as desired.

12.5.2 After obtaining that aliquot for analysis, the pH of the sample is checked using wide range pH paper. Samples should be preserved to a pH <2. Document pH on instrument run log. Notify project manager of samples with a pH >2 if not analyzed within 7 days from collection. Analysts should always obtain their sample aliquots before taking pH. If sample volumes are limited (one vial per sample) the analyst should transfer a sample aliquot to a smaller container with no headspace (scintillation vial) if additional testing may be required. For limited volume samples, the analyst should obtain the pH of the sample by pipetting a small volume of the sample across the pH strip into a waste container, otherwise the pH strip may be dipped in the sample vial and the vial discarded.
NOTE: Make a note of any sample discrepancies, i.e. unpreserved sample, any headspace, and excessive sediment in the batch case narrative and on the instrument runlog.

12.5.3 After obtaining the aliquot for analysis, waters shall be checked for residual chlorine. Residual chlorine check is similar to the pH measurement, using a chlorine test strip. Additional sample aliquots cannot be taken from the vial once the residual chlorine test has been performed. For limited sample volumes the steps outlined in 12.5.2 may be necessary to preserve sample aliquots for future testing. The presence of residual chlorine is documented in the run log as “Yes” or “No”. If

residual chlorine is present in samples, the analyst should document the information in the case narrative, and notify the project manager.

12.5.4 TCLP extracts and Tumble blanks are diluted prior to analysis for a total 5 mL prep. Transfer the 5 mL of sample to a clean 40 mL autosampler vial and place the vial in the appropriate autosampler position. Complete the autosampler sequence as desired. The batch QC requirements are a tumble blank, method blank, LCS, post spike and post spike duplicate.

13.0 QUALITY CONTROL SAMPLE FOR SOLID AND AQUEOUS SAMPLES

13.1 After preparing and prior to purging, fortify the appropriate samples for matrix spike purposes using an amount of MS/LCS spike solution needed to achieve the desired concentration.

13.2 Preserved soils (5035) should be fortified by inserting the syringe needle into the vial septum and injecting the spike solution into the liquid. Tilt the vial during this process to ensure that the spike solution enters the liquid phase of the sample.

13.3 Aqueous and/or 5035A solid samples can be fortified in the syringe prior to transferring the aqueous sample or organic free water to the autosampler vial.

13.4 Prepare the Laboratory Control Sample (LCS) for solids and liquids by injecting the appropriate amount of spike solution needed to achieve the desired concentration into a gas tight syringe containing 5 mL organic free water. Transfer the syringe contents to an empty 40 mL autosampler vial or one containing 5 g of sand.

13.5 Prepare aqueous method blanks by transferring 5 mL organic free water to an empty 40 mL autosampler vial. Prepare solid method blanks by transferring 5 mL of organic free water to a 40 mL autosampler vial containing a stir bar and 5 g of sand.

13.6 Quality control samples for solids should contain 5 g of sand and a stirbar.

14.0 OVERALL QUALITY CONTROL REQUIREMENTS

14.1 Prior to any quality control or sample analysis, the analyst must perform an instrument check by injecting 5 to 50 ng of Bromofluorobenzene (BFB). The BFB tune check must be performed prior to each analytical sequence. A new BFB check must be performed after 12 hours of analysis.

14.2 Prior to any sample analysis the instrument must be calibrated. Initial calibration standards are analyzed if the calibration verification standards do not meet the acceptance criteria or if instrument maintenance was performed (i.e., source cleaning, new column, replacing source parts).

14.3 The initial calibration must be verified in each 12-hour sequence by purging a mid-level calibration verification standard. For SC samples the calibration verification standard must come from the same source standard that is used to prepare the initial calibration curve.

14.4 A laboratory control sample must be analyzed during each 12-hour sequence by purging a second source mid-level standard.

14.5 A method blank must be analyzed during each 12-hour sequence by purging organic free water.

14.6 Matrix spike and matrix spike duplicate samples are analyzed in each batch of up to 20 samples of the same matrix. Many clients require their sample to be used for matrix spike purposes. Refer to client specifications in AlphaLIMS for this information.

14.7 A laboratory control sample duplicate should be analyzed if matrix volumes are limited.

14.8 Surrogate compounds and internal standards are added to each sample and all quality control samples.

15.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

15.1 Prior to the analysis of calibration standards, inject or introduce 5 to 50 ng of the bromofluorobenzene (BFB) standard into the GC/MS system. The mass spectra of BFB can be obtained using an average of up to three scans and using a background subtraction from no more than 20 scans before the peak starts. However, the analyst may use other documented approaches by the instrument manufacturer.

15.2 Initial Calibration. An unpreserved calibration curve may be used for high level methanol extracts, waters and low level soils. Standards at various concentrations are analyzed and the instrument responses they generate are entered into the analytical method. Traceability of calibration and calibration verification standards is documented per GL-LB-E-007. Individual identification numbers are assigned to each source standard. The calibration curve must be a contiguous subset of the original set.

NOTE: Prior to tuning or running a multi-point calibration, precautions are taken to ensure that the instrument is clean and functioning properly. Standards should be set up from low concentration to high concentration in deionized water (Refer to Appendix 1). Once the instrument methods have been established the mass range and scan time should not be readjusted.

15.2.1 Establish the GC/MS operating conditions using the following guidelines:

15.2.1.1 Mass Range: 35-300

15.2.1.2 Scan time: 0.6-2 sec/scan

15.2.2 Calibration standards are purchased at multiple concentration levels and packaged separately in sealed ampules. Upon cracking the ampule for each standard, transfer each solution into a micro reaction vessel with a mininert cap. Indicate on the vial label the unique standard identification number, date opened, and the expiration date plus the analyst's initials preparing/opening the standards.

15.2.3 Load a 10 mL gas tight syringe with 5mL of organic-free water. Transfer 5 µL of calibration standard into the syringe and then transfer to a 40 mL vial. Repeat this step for each concentration level of standard. The Calibration Long List is separated into gases and non-gas compounds. The analyst must add each set of concentration levels to a syringe in order to analyze for all the compounds in this list. Similarly, the Calibration Short List is prepared by adding 5 µL of the Short List standard plus 5 µL of the

Acrolein/Cyclohexanone standard into a syringe containing 5 mL of deionized water.

15.2.4 Set up the autosampler to analyze each standard from low to high concentration.

15.2.5 Tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to its internal standard. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = (AxCis) / (AisCx)$$

Where:

Ax = Area of the characteristic ion of the compound being measured.

Ais = Area of the characteristic ion of the specific internal standard.

Cis = Concentration of the specific internal standard.

Cx = Concentration of the compound being measured.

15.2.6 For 8260B the average RF must be calculated for each compound. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for minimum average response factors. These compounds are: Chloromethane, 1,1-Dichloroethane, Bromoform, 1,1,2,2-Tetrachloroethane, and Chlorobenzene. The minimum acceptable average RF for these compounds is: 0.1 for Chloromethane, 1,1-Dichloroethane, and Bromoform, 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane. These compounds typically have RFs of 0.4 to 0.6 and are used to check compound instability and degradation caused by contaminated lines or active sites in the system. Failure to meet the response factor criteria may be attributed to one or more of the following examples:

- Chloromethane: This compound is the most likely compound to be lost if the purge flow is too fast.
- Bromoform: This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve Bromoform response.
- 1,1,2,2-Tetrachloroethane and 1,1-Dichloroethane: These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

NOTE: Non-SPCC compounds should have a minimum response factor of 0.01, with the exception of poor purging compounds such as those listed in Appendix 6. Unless otherwise required by client

contract, these compounds may have response factors as low as 0.001. For the analysis of ketones and carbon disulfide at a PQL less than 5 ppb, the relative response factor for these analytes must be equal to or greater than 0.05. Method 8260C has additional requirements for response factors. Refer to Appendix 5. Method 8260D also has additional guidance for response factors however, it is not required.

15.2.7 Calibration curves must be verified using an initial calibration verification standard (ICV). This should be a second source standard from the initial calibration. The laboratory control spike solution can be used for this standard. The response factor or true value (percent difference or drift) is evaluated based on individual client requirements. At minimum the SPCC and CCC's criteria for response and percent drift or difference must be met. For samples from within the state of South Carolina the percent drift or difference should not exceed +/- 30% for requested analytes and +/- 40% for poor purgers (See Appendix 6). The percent drift or difference for non-CCC compounds may be as high as 60% for some clients. For DoD clients the percent drift or percent difference should not exceed ± 20% for requested analytes. If the ICV does not meet the criterion, corrective action should be taken (i.e., inject a different second source, open a new standard mix, perform instrument maintenance and/or reanalyze the initial calibration). If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative. See Appendix 4 for 8260C specific criteria. See Appendix 9 for 8260D criteria.

15.2.8 All newly generated calibration curves shall have a secondary review by a Group Leader or Data Validator before being used.

15.2.9 The internal standard responses and retention times in the calibration standards must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds during the calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the Extracted Ion Current profile (EICP) area for any of the internal standards changes by a factor of two (-50% to +100%) during the calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made reanalysis of the calibration check standard is necessary.

15.2.10 Using the $\overline{RF}$ s from the initial calibration, calculate the percent relative standard deviation (%RSD) for Calibration Check Compounds (CCCs) and all target analytes:

$$\%\text{RSD} = \frac{\text{SD}}{\bar{x}} \quad 100$$

Where:

RSD = Relative Standard Deviation

$\bar{x}$ = Mean of initial RFs for a compound

SD = Standard deviation of average RFs for a compound

$$SD = \sqrt{\sum_{i=1}^{n} \frac{\left(x_i - \bar{x}\right)^2}{n-1}}$$

15.2.11 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range and may be used for quantitation. If the RSD of any target analyte is greater than 15%, then use one of the options in section 15.2.13. A minimum of five calibration standards must be used. The laboratory may use more than five concentration levels. The maximum % RSD for NNSA client is 60% Refer to Appendix 4 for 8260C and Appendix 10 for 8260D

15.2.12 The % RSD for each individual CCC must be less than or equal to 30 percent difference. This criterion must be met in order for the individual calibration to be valid. The CCCs are:

- 1,1-Dichlorethane
- Chloroform
- 1,2-Dichloropropane
- Toluene
- Ethylbenzene
- Vinyl Chloride

15.2.13 Given the potentially large numbers of analytes that may be analyzed by this method, it is likely that some analytes may exceed that 15% acceptance limits for RSD. In those instances, the following steps may be used:
NOTE: Refer to Appendix 4 and 10 for 8260C criteria.

15.2.13.1 Check the instrument operating conditions. Follow the suggested maintenance procedure in this method to make minor adjustments to the system.

15.2.13.2 Review the results of each standard (area counts, calibration or response factors and RSD) to ensure that the problem is not associated with one of the five initial calibration standards. If the problem appears to be associated with a single standard, then the entire calibration must be reanalyzed.

15.2.13.3 High-end standards may be removed on a compound by compound basis thus narrowing the calibration range of the instrument if there are enough remaining calibration points to meet method requirements. The top of the calibration curve may

not be the mid-point spiking concentration. At least one point must be in the calibration curve above this concentration.

15.2.13.4 The analyst may also choose to construct calibration curves of area ratios versus concentration using first order regression fit of the five calibration points. The regression calculation will generate a coefficient of determination (r2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r2 must be ≥ 0.990. The minimum acceptable correlation coefficient (r) for any compound using linear regression shall be 0.995. The analyst will select only linear regression or average response factor. Higher order curves such as a quadratic curve must never be used. Correlation coefficients may not be rounded up to meet these requirements.

15.2.13.5 Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where:

y = instrument response

a = slope of the line (also called the "coefficient of x")

x = concentration of the calibration standard

b = the intercept

15.2.13.6 If the analyst chooses to use linear regression, he/she must not force the calibration line through the origin but have the intercept calculated from the data points. In addition, do not include the origin (0,0) as a sixth calibration point.

15.2.13.7 The calculated intercept value needs to be evaluated before reporting sample results. A positive value for the intercept indicates that there is some threshold instrument response that is the limiting factor in establishing linearity. A negative intercept value can be transformed into an x-intercept value that represents a threshold concentration that is the limitation. If the intercept is positive, then as a general rule, results where the instrument response is less than three (3x) the intercept value may be unreliable. This will afford some protection against false positive results. If the intercept is negative, results below the concentration of the lowest concentration calibration standard may be unreliable. The value of the y intercept multiplied by the internal standard concentration must be less than 3 times the

value of the MDL for each matrix for analysis of NNSA client samples. A weighted linear calibration curve may be generated and the intercept re-evaluated for NNSA requirements.

15.2.13.8 In calculating the sample concentrations, the regression equation is rearranged to solve for the concentration (x):

y = ax + b: linear equation

x = (y-b)/a

15.2.14 Exclusion of initial calibration points without technical justification is not allowed. Occasionally, it may be obvious that a condition such as a bad injection or purge had rendered an initial calibration standard unusable.

15.2.14.1 If it is necessary to replace a calibration level in a curve, the entire level must be replaced and not individual analytes.

15.2.14.2 The questionable level may be reanalyzed as long as the reanalysis is performed within the 12 hour window. The second injection must be used in the calibration curve.

15.2.14.2.1 Should the second injection fail, the entire calibration curve must be reanalyzed.

15.2.14.2.2 If the second injection passes, reanalyze the initial calibration verification sample.

15.2.15 Method 8000D outlines two procedures that may be used to determine calibration function acceptability for linear and non-linear curves. These include refitting the calibration data back to the model. % Error and Relative Standard Error (RSE) evaluate the difference between the measured amount and the true amount (or concentration).

15.2.16 % Error is determined as follows:

$$\%Error = \frac{x_i - x_i'}{x_i} \times 100$$

Where:

x′i= Measured amount of analyte at calibration level i, in mass or concentration units

xi= True amount of analyte at calibration level i, in mass or concentration units.

15.2.17 Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤ 50% may be acceptable for the lowest concentration point.

15.2.18 Relative Standard Error is calculated as follows:

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i} \right]^2 \Big/ (n - p)}$$

Where:

xi= True amount of analyte in calibration level i, in mass or concentration units

x′i= Measured amount of analyte in calibration level i, in mass or concentration units

p= Number of terms in the fitting equation (average =1, linear =2, quadratic =3, cubic =4)

n= Number of calibration points

15.2.19 The RSE acceptance limit criterion for the calibration model is the same as the RSD limit in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤ 20% for good performing compounds and ≤ 30% for poor responding compounds.

15.3 Continuing Calibration Verification

15.3.1 Prior to the analysis of calibration verification standards, inject or introduce 5 to 50 ng of the Bromofluorobenzene standard into the GC/MS system. The resulting mass spectra of the BFB must meet all of the criteria given in Appendix 2 before sample analysis begins. The mass spectrum of BFB can be obtained using an average of up to three scans and using a background subtraction of a single scan no more than 20 scans before the peak starts. However, the analyst may use other documented approaches suggested by the instrument manufacturer.

15.3.2 The initial calibration curve for each compound of interest must be checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a calibration standard that is at a concentration near the mid-point concentration for the working range of the GC/MS by checking SPCCs and CCCs. This will determine the validity of the initial calibration on a daily basis. The continuing calibration check standard may be a second source standard. For SC samples the CCV must come from the same source standard that is used to prepare the initial calibration curve. Refer to Appendix 4 for 8260C and Appendix 8 for Standard Method 6200 specific criteria and Appendix 10 for 8260D for specific criteria.

15.3.3 System Performance Check Compounds (SPCCs): A calibration verification check must be made every 12 hours. If the SPCC criteria are met a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. The minimum response factors for volatile SPCCs are listed in section 15.2.6 of this SOP. Some possible problems that may prevent one from meeting the criteria are outlined in that section.

15.3.4 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs are used to check the validity of the initial calibration. Calculate the percent difference using:

$$\%\text{Difference} = \frac{\left|\overline{RF_I} - RF_c\right|}{\overline{RF_I}} \times 100$$

Where:

$\overline{RF_I}$ = Average response factor from initial calibration
RFC = Response factor from current verification check standard

15.3.5 If the percent difference for all CCCs is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% difference), corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new calibration will be analyzed.

15.3.5.1 Passing CCC and SPCC criteria should not be the only factors in evaluating a successful calibration verification. All the analytes in the standard should be evaluated before analysis continues. The percent drift or difference for non-CCC compounds may be as high as 60% for some clients. For DOD-QSM clients the percent drift or percent difference should not exceed 20% for requested analytes. For samples from within the state of South Carolina the percent drift or difference should not exceed ± 30% for requested analytes and ± 40% for poor purging compounds (See Appendix 6). If an analyte is flagged as an outlier on the report the analyst should at least evaluate the significance of the flag. The reason for the outlier may be as simple as the computer missing a quant ion, a poor performance compound, or loss of moisture control. Whatever the reason for the flag, all outliers should be evaluated on how the data will be affected by the analyte list. The 20% criteria for CCCs may be waived if all the required target analytes are less than 20% difference/drift. In addition, if the CCC and SPCC criteria are met despite these compounds not being required, the required target analytes may have differences/drifts greater than 20%. Again, every standard should be evaluated carefully in order to obtain quality data. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative. Refer to Appendix 4 for 8260C and Appendix 8 for Standard Method 6200 specific criteria and Appendix 10 for 8260D specific criteria.

15.3.5.2 Internal Standard Response: Internal standard areas in the daily continuing calibration verification standard (CCV) must be within +100 to -50% and retention times for each internal

standard must be within ±30 seconds of the initial calibration midpoint internal standard. If internal standards do not meet these criteria, reanalyze the CCV to verify the problem was not random. If the areas fail again, corrective action must be taken. Several actions may correct the problem, i.e., refreshing the internal standard solution on the autosampler, using a newly opened solution, checking that instrument conditions have not changed, etc. Document the actions taken to correct the problem in the instrument maintenance log. Refer to Appendix 4 for 8260C specific criteria.

15.3.5.3 If the continuing calibration does not meet the aforementioned criteria, the analyst must determine why and take appropriate corrective action. Samples may not be analyzed until the validity of the calibration has been verified.

16.0 ANALYSIS PROCEDURES AND INSTRUMENTAL OPERATION

16.1 All calibration and verification standards, blanks, quality control samples, and client samples are analyzed using a heated purge. Ultra Pure helium is bubbled through the sample that is contained in a purging chamber at 40° C. The purgables are efficiently transferred from the aqueous phase to the vapor phase that is swept through a sorbent where purgeables are trapped. After purging is complete, the trap is heated and backflushed with the inert gas to desorb the analytes onto a gas chromatograph that is temperature programmed to separate the analytes that are then detected with a mass spectrometer.

16.2 Daily GC/MS Analysis Procedure:

At the beginning of each day or 12-hour tune window, the GC/MS system is checked to see if acceptable performance criteria are achieved. A 5 to 50 ng BFB standard is analyzed and must pass acceptance criteria before any further analyses are performed.

16.3 If the tune passes, a continuing calibration verification sample is analyzed. At a minimum the SPCC and CCC criteria for response factor and percent drift must be met. Additionally for samples within the state of South Carolina, the percent drift or difference should not exceed +/- 30% for the requested target analytes and +/- 40% for poor purgers (See Appendix 6). If the client requested target list does not include SPCC or CCC compounds, all compounds must have a percent difference or drift of less than 20%. For client samples outside of the state of South Carolina, the CCV should be evaluated against client specific requirements if available. The percent drift or difference for non-CCC compounds may be as high as 60%. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative. Refer to Appendices 4 and 5 for 8260C and Appendix 8 for Standard Method 6200 specific criteria and Appendix 10 for 8260D specific criteria.

16.4 Upon passing the calibration verification, a laboratory control sample (LCS) is analyzed. The LCS must pass either the laboratory SPC limits or client specific limits before

continuing analysis. The CCV and LCS may be combined into one analysis as long as the standard is a second source to the initial calibration standards. The standard must pass both sets of criteria for the CCV and LCS to be acceptable. For SC samples the CCV must come from the same source standard that is used to prepare the initial calibration curve and the LCS must come from a second source standard. Some clients may require that the laboratory analyze a CCV and LCS as two separate injections, specific client requirements are indicated on the batch pull sheet. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative.

16.5 Upon analysis of an acceptable LCS(s), the analyst must analyze an acceptable method blank prior to beginning client samples.

16.6 Upon the analysis of an acceptable method blank, client sample analysis may begin. Analysts are encouraged to begin the sequence with the analysis of samples that should be clear of contamination such as trip blanks, field blanks, and methanol blanks.

16.7 If a sample contains target and/or non-target analyte concentrations that exceed the calibration range, the analyst must review subsequent sample analyses for possible carryover contamination of those analytes. Samples containing suspected carryover contamination are reanalyzed. The system is decontaminated either by the analysis of subsequent blanks or subsequent clean samples.

16.8 Sample analysis may continue up to 12 hours from the injection time of the BFB. Any sample analyzed after the 12 hour window will require reanalysis in a valid 12 hour sequence.

16.9 Each instrument has a run log in which each 12 hour sequence is documented. The analyst must document standards used in the analysis, in addition to data files, dates, injection times, sample weights, volumes, pH, residual chlorine, and additional comments.

17.0 QUALITY CONTROL REQUIREMENTS

17.1 Frequency of Quality Control Activities

17.1.1 Instrument Check

Before any initial calibration or continuing calibration checks are performed, the analyst must analyze an acceptable Bromofluorobenzene (BFB) solution (5 to 50ng). Refer to Appendix 2 or 9 for criteria.

17.1.2 Initial and Continuing Calibration

Before the analysis of any blanks, spikes or samples, the instrument must be calibrated. This multilevel calibration is used to establish the linearity of the instrument for the analytes of interest. Once this calibration is in place, the frequency thereafter is determined from the analysis of continuing calibration standards.

17.1.3 The continuing calibration standard is used to verify instrument calibration compared to the last multilevel calibration check and must be performed every 12 hours.

17.1.4 Blank Analysis

17.1.4.1 Method Blanks are used to determine background (laboratory) concentrations of target analytes that have the potential of interfering with sample analysis. A blank is analyzed in each 12-hour analytical sequence.

17.1.4.2 Other types of blanks can be used to determine possible contamination from various points in the process. Trip Blanks can be used for evaluating the bottles in which the samples are collected. Field Blanks are used to discover potential interferences that may originate from the collection process. The frequency at which such blanks are analyzed are determined by the client (internal or external). Refrigerator or Storage Blanks are analyzed on at least a biweekly basis to determine the cleanliness of the storage area.

17.1.5 Spike and Duplicate Analysis

17.1.5.1 Accuracy is monitored through the analysis of sample spikes. Matrix spikes are performed for each batch up to maximum of 20 samples of similar matrix.

17.1.5.2 Precision is monitored through the analysis of duplicates. For method 8260B/C, the matrix spike or sample is duplicated. This ensures the availability of two sets of results for comparison.

17.1.5.3 Laboratory Control Samples (LCS) are used to determine if the process is in control. If a matrix spike fails due to matrix related interference, then the analysis of the LCS demonstrates that the instrument is in control. For non-SC samples, the continuing calibration check may be used as an LCS if it is a second source standard. Analyze the LCS in each 12-hour sequence. The LCS may be analyzed before the method blank as the CCV. For SC samples the LCS must come from a second source standard.

17.1.6 Surrogate Spikes must be added to all standards, blanks, quality control, and client samples.

17.1.7 Internal standards must be added to all standards, blanks, quality control, and client samples.

17.1.8 Samples must be diluted whenever a target analyte concentration exceeds the highest concentration (for that analyte) in the calibration curve. Samples should be diluted in order to put the analyte concentrations near or above the midpoint of the calibration curve. Multiple dilutions may be necessary if multiple analytes at various concentrations require dilutions. Also, if a sample is initially diluted and there are no target or non-target interferences present, the analyst may be required to reanalyze a more concentrated sample. Some factors for consideration when choosing a dilution factor are

pH, nature of the matrix and non-target interferences. Analysts should be aware that diluting samples increases the detection and reporting limits.

17.2 Acceptance Limits for Quality Control Activities

17.2.1 Initial Calibration

17.2.1.1 The initial calibration %RSD and RRF calculated for each compound must meet the criteria in section 15 of this SOP. Refer to Appendix 4 and Appendix 5 for 8260C specific criteria.

17.2.1.2 The internal standard areas and retention times in each calibration standard analysis must meet the criteria in section 15 of this SOP. Refer to Appendix 4 for 8260C and Appendix 10 for 8260D criteria Appendix 8 for 8260D for Standard Method 6200.

17.2.1.3 The initial calibration must be verified by the analysis of an acceptable ICV before client samples can be analyzed. Refer to Appendix 4 for 8260C and Appendix 10 for 8260D criteria.

17.2.2 Continuing Calibration

17.2.2.1 The instrument calibration must be verified every 12 hours by analyzing an acceptable continuing calibration verification standard (CCV). The CCV must meet the criteria indicated in section 15. Refer to Appendix 4 and Appendix 5 for 8260C, and Appendix 8 for Standard Method 6200 specific criteria and Appendix 10 for 8260D criteria.

17.2.3 Method blank criteria require that concentrations of all analytes of interest must be below the client reporting limit or laboratory practical quantitation limit. Ideally, all target analyte concentrations should be below the laboratory method detection limit, however, concentrations below reporting limits are acceptable. Specific client criteria may require that target analyte concentrations in the method blank not exceed one half the reporting limit. The analyst should take corrective action to ensure that target analytes do not exceed the reporting limits. Corrective action may include the analysis of clean up blanks or perhaps, more intense system decontamination. In some cases, concentrations for the common laboratory contaminants (methylene chloride, acetone, 2-butanone, ethyl ether, carbon disulfide) may be present in the method blank above the reporting limit, but the concentration(s) must be less than five times the reporting limit. In this situation if the compounds are detected in the associated samples, the data are flagged and notation is made in the case narrative that detection and quantitation of these analytes in samples would be biased. If the compounds are not detected in the associated samples, no action is required.

17.2.4 Matrix Spikes/Duplicates

17.2.4.1 SW-846 8260C/B/D criteria for both matrix spikes and matrix spike duplicates will be statistically determined after at least 20 to 30 matrix pairs. If data are not available for specific compounds,

a guidance of 70% to 130% recovery for spikes may be used. Some clients may have contract required limits that the laboratory should use.

17.2.4.2 Target analyte concentrations in the parent sample that exceed five times the spike level may cause biased recovery results in the matrix spike samples. Unacceptable recoveries due to high concentrations in the parent sample should be noted in the case narrative.

17.2.4.3 The criteria for sample duplication when analyzing samples using method 8260C/B/D are that any target present over five (5) times the detection limit must be within ± 20% of each other. As a statistical database is built, statistical process control (SPC) limits are generated.

17.2.4.4 Laboratory control sample recoveries must be within the established SPC limits to be considered acceptable. Statistical control limits for each compound should be derived after the analysis of up to 20 to 30 LCSs (per matrix). The SPC limits for most volatile analytes should fall between 70% to 130% recovery; however, due to poor purging efficiencies, water solubilities, and poor chromatography, some analytes may not meet the method recommendation.

17.2.4.5 Surrogates in all quality control and client samples must meet the established SPC limit or client specified limits. Surrogate limits are determined statistically at least semi-annually utilizing a minimum of 20 data points. Samples must be reanalyzed if surrogate recoveries are outside the required limits. If the surrogate recoveries are unacceptable in the reanalysis, the failure is attributed to matrix. One or both analyses may be reported depending on client specifications.

17.2.4.6 Internal standard responses must be within the required criteria in the initial calibration and calibration verification standard (Section 15). In addition to the internal standard area/RT check from the calibration verification standard to the midpoint of the current initial calibration, the laboratory also compares the internal areas and retention times of samples and QC to the associated daily calibration verification standard. If area counts exceed –50% to +100% or retention times are greater than ± 30 seconds, the sample or QC sample must be reanalyzed. If the analyst suspects matrix interference the sample may be diluted to aid in the internal standard recovery. If the response for the internal standards is unacceptable in the reanalysis, the failure is attributed to matrix. One or both analyses may be reported depending on client specifications.

18.0 ANALYST AND METHOD VERIFICATION REQUIREMENTS

18.1 New analysts must complete an Initial Demonstration of Proficiency prior to analyzing client samples. The analyst must be able to demonstrate proficiency by analyzing a passing tune, calibration verification, laboratory control samples, and method blank. The initial demonstration is documented using four acceptable LCS analyses in an analytical sequence.

18.2 Continuing Demonstration of Proficiency results are generated by collecting the data from four LCS analyses per method per analyst. The QA/QC group generates the certificates annually. Refer to GL-QS-E-011 Method Validation and Initial and Continuing Demonstration of Capability.

18.3 Method Detection Limits (MDL) are analyzed annually and verified quarterly. Refer to GL-LB-E-001 for The Determination of Method Detection Limits for additional information.

19.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

19.1 Routine Preventative and Special Operational (Failure)

19.1.1 Preventative maintenance on a GC/MS system involves four basic areas:

19.1.1.1 Vacuum pumps for the analyzer should have an oil change when the system demonstrates that it is needed. This includes the mechanical, turbomolecular, and/or diffusion pumps. Generally, this occurs at least once a year.

19.1.1.2 GC maintenance consists mainly of column and injection port maintenance. These procedures are performed when system sensitivity drops, or air (m/z = 28) is noted present in the system.

19.1.1.3 Analyzer maintenance is most often usage dependent. The type and quantity of samples that have been injected, determine the frequency of ion source and electron multiplier cleaning and/or replacement.

19.1.1.4 Autosampler maintenance is very sample dependent. In general, maintenance consists of changing the trap and line cleaning. Line cleaning is accomplished by back flushing the system with methanol.

19.1.2 Non-routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

19.1.2.1 Troubleshooting the autosampler and instrument is a function of analyst experience. In-House service is obtained from GEL's Service Technician. If vendor assistance is needed, then the appropriate vendor is contacted.

19.1.2.2 Electronic maintenance logbooks are kept for each instrument and include entries for both routine as well as non-routine maintenance procedures.

20.0 DATA RECORDING, CALCULATIONS AND REDUCTION METHODS

20.1 Qualitative Analysis:

20.1.1 Samples, standards, and quality control samples are processed using HP Chemstation software. The software performs identification and quantitation based off method information applied to the raw data areas or responses. The program generates electronic reports for review and validation. Raw data are stored electronically on servers and transferred to compact disc media.

20.1.2 Compound Identification

20.1.2.1 A comparison of the sample mass spectrum with the mass spectrum of a standard reference may identify the suspected compound. Mass spectrum for standard reference must be obtained from the NIST library.

20.1.2.2 Elution of sample component at the same GC relative retention time (RRT) as those of the standard component may help identify a compound.

20.1.2.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

20.1.2.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two peaks is less than 25% of the sum of the heights. Otherwise, structural isomers are identified as isomeric pairs.

20.1.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample, appropriate selection of analyte spectra and background spectra is important.

20.1.2.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in the qualitative identification of compounds. When analytes coelute, the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

20.1.2.7 Occasionally manual integrations may be required if the sample matrices affect the software's ability to draw appropriate baselines. Manual integrations on calibration levels are permitted although discouraged. In the event that a manual integration is

performed, the software generates a printout of the integration event before the manual integration and after the manual integration. The analyst electronically reviews and initials the data file and includes the reasoning behind the manual integration.

20.1.3 The relative retention time (RRT) of the sample component must be within ± 0.06 RRT units of the RRT of the standard component, analyzed in the same 12 hour period. If coelution of another compound prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the components of interest. Analysts should pay close attention to the following compounds for possible coelution and identification 1,1,1-Trichloroethane, and Carbon tetrachloride; m,p-Xylenes and Ethylbenzene; the three Dichlorobenzene isomers; Chloromethane and Dichlorodifluoromethane, cis-1,2-Dichloroethylene and trans-1,2-Dichloroethylene; cis-1,3-Dichloropropylene and trans-1,3-Dichloropropylene; 2-Chlorotoluene and 4-Chlorotoluene; the two Trimethylbenzenes; the three Butylbenzenes; and the two Trichlorobenzenes.

20.1.4 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analysis being conducted. Refer to Appendix 7 for COA and Form 1 TIC reporting.

20.2 Quantitative Analysis:

20.2.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of a given analyte. The concentration of each identified analyte in the sample is calculated as follows:

20.2.2 Water and Water- Miscible Waste using Average RRF:

$$\text{Concentration } (\mu g/L) = [(Ax)(Cis)(Vo)] / [(Ais)(RRF)(Vi)]$$

Where :

Ax =	Area of characteristic ion of compound being measured
Cis =	Concentration of internal standard purged in µg/L
Ais =	Area of Characteristic ion for internal standard

RRF= Relative response factor for compound being measured (see above)

Vo = Total purge volume (mL)

Vi = Volume of sample purged (mL)

20.2.3 Water and Water-Miscible using Linear Regression:

$$\text{Conc. } \mu g/L = \frac{\left[\frac{\left(\frac{Ax}{Ais}\right)}{a} + b\right] * Cis * Vo}{Vi}$$

Where:

Ax = Area of characteristic ion of compound being measured

Cis = Concentration of internal standard purged in µg/L

Ais = Area of characteristic ion for the internal standard

RRF = Relative response factor for compound being measured (see above)

Vo = Total purge volume (mL)

Vi = Volume of sample purged (mL)

a = slope

b = y intercept

20.3 Sediment, Soil, Sludge, and Waste

20.3.1 High level using Average RRF:

Concentration (µg/kg) = [(Ax)(Cis)(Vt)(Vo)] /[(Ais)(RRF)(Ws)(Vi)]

20.3.2 Low Level using Average RRF:

Concentration (µg/kg) = [(Ax)(Cis)(Vt)(Vo)] /[(Ais)(RRF)(Ws)(Vi)]

Where :

Ax, Cis, Ais, RRF = Same as for water

Vt = Volume of total extract (use 10,000 µL or factor of this when dilutions are made.)

Vi = Volume of extract added (µL) for purging

Ws = Weight of sample extracted or purged (g). The wet or dry weight may be used, depending upon the specific application of the data

Vo = Total purge Volume (mL)

20.3.3 Low Level using Linear Regression:

$$\text{Conc. } \mu g/kg = \text{Conc. } \mu g/kg = \frac{\left[\frac{\frac{Ax}{Ais}+b}{a}\right] * Cis * Vo}{W_S}$$

20.3.4 High Level using Linear Regression:

$$\text{Conc. } \mu g/kg = \text{Conc. } \mu g/kg = \frac{\left[\frac{\frac{Ax}{Ais}+b}{a}\right] * Cis * Vo * Vt}{Ws * Vi}$$

20.4 Where applicable, an estimate of concentration for non-calibrated components in the sample is made. The formulas given above shall be used with the following modifications: The areas Ax and Ais shall be from the total ion chromatograms, and the RF for the compound shall be assumed to be 1.

20.4.1 The concentration obtained is an indication that the value is an estimate, and identifies which internal standard was used to determine concentrations. Use the nearest internal standard free of interferences.

20.5 SPC limits are determined by criteria set forth in EPA Method 8000D. For a given matrix, calculate the upper and lower control limit for method performance for each standard of interest as follows:

Upper control limit (UCL) = p + 3 s

Lower control limit (LCL) = p - 3 s

Where:

p = Average percent recovery

s = Standard deviation of percent recovery

20.6 Data are reported in units of ug/L or ppb for waters, and ug/kg or mg/kg for soils and sludges unless otherwise specified by the client.

21.0 POLLUTION/CONTAMINATION CONTROL

21.1 The volatile organics laboratory is physically separated from the other areas of the laboratory. Only authorized personnel have access to this area.

21.2 A separate air handling system and frequent filter changes help keep this area free of common contaminants. The laboratory includes a positive pressure air system.

21.3 Volatile samples are stored separately from other samples. Oil or waste samples for volatile organic analysis are generally not stored in the volatile refrigerator. Samples that may cause contamination in the refrigerator are stored in the main walk-in cooler.

Storage blanks are analyzed at least biweekly from each refrigerator and cooler that stores samples for volatile analysis.

21.4 Soils and waste samples are weighed on a balance within a fume hood in the volatile organics laboratory.

21.5 Radioactive and non-radioactive samples are separated in different refrigerators.

21.6 Organic free water obtained by passing laboratory tap water through a series of carbon filters. This system is located in the volatile organics laboratory.

22.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

22.1 After analysis, each analyst is responsible for reviewing their analytical data for completeness and quality control information.

22.2 The analyst electronically dates and initials the front page of the quantitation report generated by the Chemstation software.

22.3 Occasionally, manual integrations may be required if the sample matrices affected the software's ability to draw appropriate baselines. Manual integrations on calibration levels are permitted although discouraged. In the event that a manual integration is performed, the software generates a printout of the integration event before the manual integration and after the manual integration. The analyst electronically initials and dates the printout and includes the reasoning behind the manual integration.

22.4 After the analyst has completed and reviewed the batch raw data, a second review by a peer or validator is performed before data are given a done status. Data deliverables are client specific, however if a fractional case narrative of CLP-like data package is needed, an additional review of the fractional data package is performed by a validator prior to sending to the client.

22.5 The levels of review responsibility are:

22.5.1 First level review: The analyst must ensure the overall quality of the data. Included in this review are: the BFB tune criteria, samples within tune time, calibration verification standard check, method blank, matrix spikes, laboratory control sample, surrogate recovery, internal standard response, over range concentrations include a dilution, reanalysis as required, manual integrations are appropriate and correct.

22.5.2 Second level review: A peer must review all of the data before it can be released from the lab. The peer performs all the checks that the first analyst did and compares the data with the runlog. Particular attention must be paid to the 12-hour tune window, the check standard, and internal standard area counts. All hits must also be reviewed very carefully. When the second level review is complete, the peer reviewer initials and dates the quantitation report.

22.5.3 A flow chart showing the process by which papers and documents undergo review can be viewed in Figure 2.

22.5.4 Data packages are generated electronically in Portable Document Format or PDF. Data packages are sent directly to the client as a PDF and/or hard copies are generated for submittal.

22.5.5 Data packages are stored on compact disc (CD) by the laboratory computer services group.

23.0 NONCONFORMANCE AND CORRECTIVE ACTION FOR UNACCEPTABLE DATA

23.1 When analyzing a multi-point calibration for many analytes at once, the chances are high that a few may not meet the criteria. Additional standards may be reanalyzed. If they still do not meet the requirements, the instrument may need maintenance before continuing or new standards may be needed.

23.2 If the continuing calibration fails any of the criteria in section 15, the analyst must take action to correct the situation. This may be retuning, reanalyzing the standard, adjusting the purge flow, or any number of maintenance practices in order to meet the daily calibration. If all attempts fail, the analyst must analyze a new series of multi-point calibration standards. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative.

23.3 When the method blank fails the criteria defined in section 17.2.3, the analyst must find and eliminate the source of contamination before proceeding with analyses. Another blank must be analyzed before sample analysis begins. Note any nonconformance in the case narrative.

23.4 If any surrogate recovery is outside the acceptance criteria, the sample must be reanalyzed. If the surrogate recoveries fail a second time, in the same manner, and the blank and LCS recoveries do not indicate a system problem, the failure is attributed to matrix effects. Note any nonconformance in the case narrative.

23.5 All analyte recoveries in the LCS should meet either the SPC limits or client required limits. If the LCS recoveries are outside the acceptance criteria, the analysis should stop and corrective actions should be attempted before continuing with the analysis. Corrective action may be repeating the analysis, opening a new spike mix and reanalyzing, or more complex actions may be attempted. If re-tuning the instrument, the analyst must analyze a tune check (BFB) and calibration verification standard before reanalyzing the LCS. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative.

23.6 The manner in which data will be accepted or rejected is described in the flowchart in figure 1.

24.0 RECORDS MANAGEMENT

24.1 Data generated as a result of this procedure are stored in the laboratory for approximately 1 month.

24.2 After approximately 1 month these records are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

25.0 LABORATORY WASTE HANDLING AND DISPOSAL

25.1 All organic waste solvents, extracts and reagents are stored in a certified hazardous waste can. When the can is full, the GEL hazardous waste disposal specialist is called. Refer to the Laboratory Waste Management Plan (GL-LB-G-001).

26.0 REFERENCES

26.1. Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1(Part 2) Section B. SW-846, Third Edition. Method 8260B, "Volatile Organic Compounds by Gas Chromatograph/Mass Spectrometer (GC/MS): Capillary Column Technique," Revision 1, September 1994. USEPA Office of Solid Waste and Emergency Response, Washington, DC 20460.

26.2. Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods, Volume 1(Part 2) Section B. Sw-846, Third Edition. Method 8000D, "Determinative Chromatographic Separations, "Revision 4, July 2014. USEPA Office of Solid Waste and Emergency Response, Washington, DC 20460.

26.3. Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1(Part 2) Section B. SW-846, 3rd Edition. Method 5030B, "Purge-and-Trap for Aqueous Samples," Revision 2, December 1996. USEPA Office Of Solid Waste and Emergency Response, Washington, DC 20460.

26.4. Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1(Part 2) Section B. SW-846, Third Edition. Method 5035, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Revision 0, December 1996. USEPA Office Of Solid Waste and Emergency Response, Washington, DC 20460.

26.5. Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.3, July 2019.

27.0 HISTORY

Revision 24: Added LOQ and LLOQ definitions. Removed reference to 8000C 5030C. Updated the data reviewed electronically. Updated Equipment section with specific vials used. Updated calibration short and long list tables.

Revision 25: Added clarification on the frequency at which LLOQs are verified and that the LLOQ is verified on every instrument where data is reported.

Revision 26: Updated 26.2 to reference current revision number and revision month.

Revision 27: Updated to include 8260D. Updated reference section for current DoD QSM Version 5.2, December 2018

Revision 28: Updated the preservation requirement for Acrolein and Acrylonitrile to a 4-5 pH and analyzed within 7 days of sampling. Add the requirements for TCLP and tumble blanks. Updated reference for DOD QSM Version 5.3 July 2019.

FIGURE 1: DECISION FLOWCHART

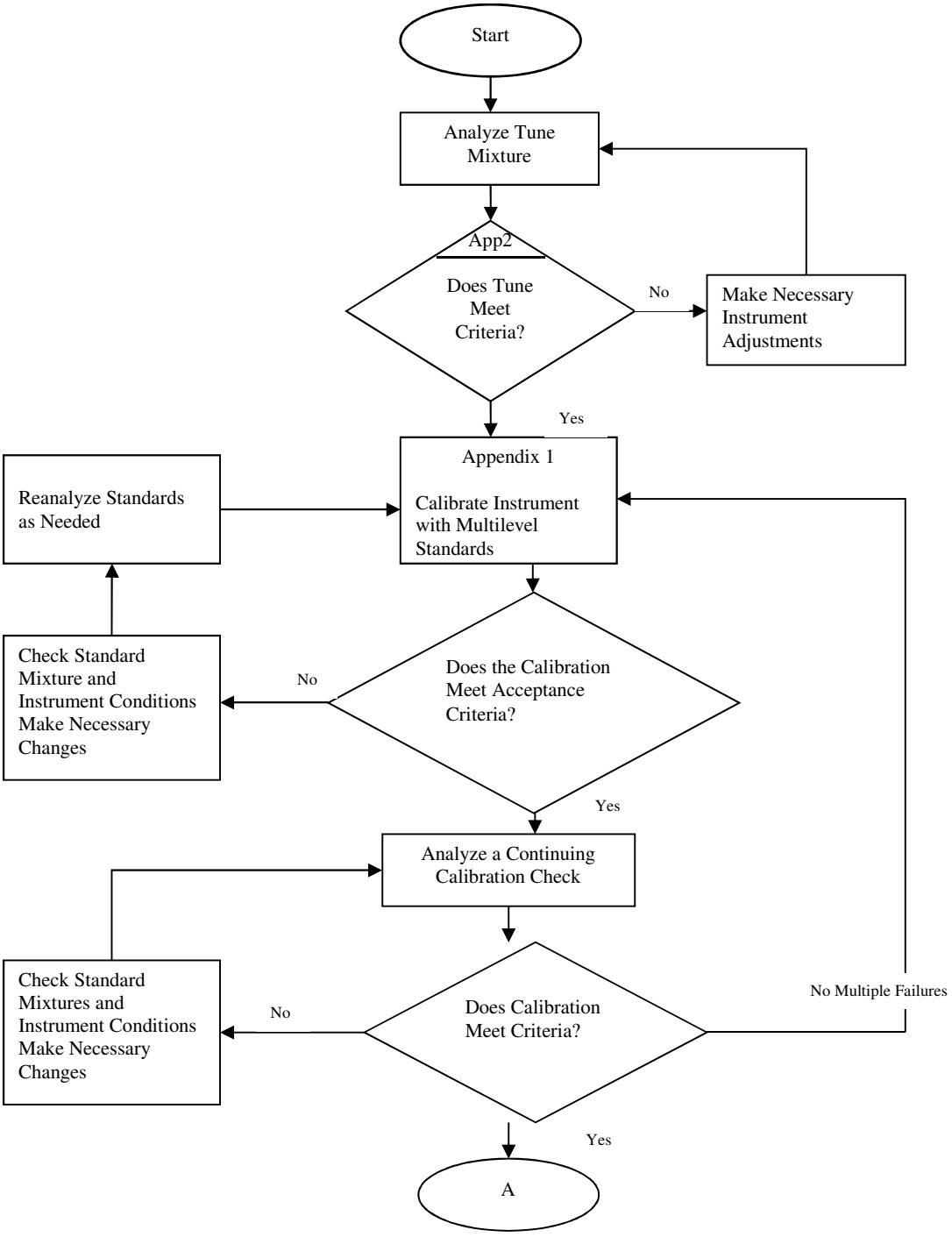


FIGURE 1: DECISION FLOWCHART continued

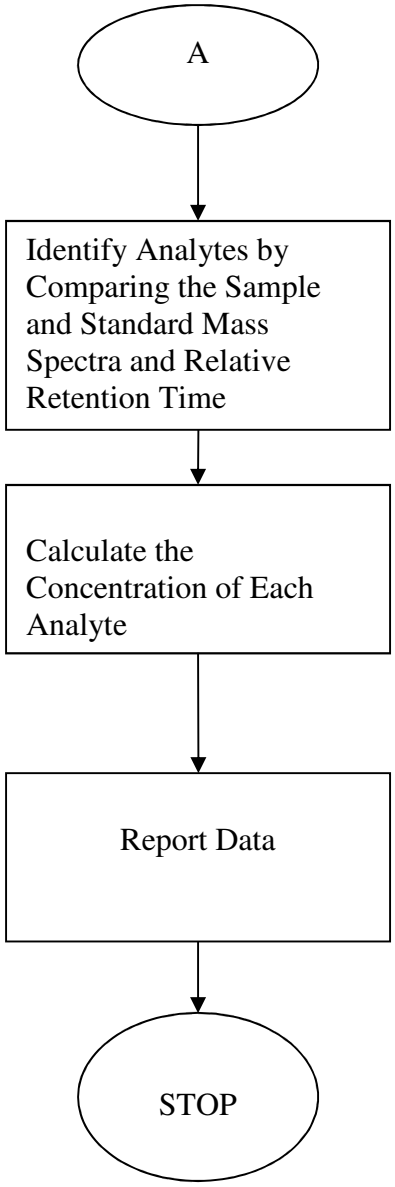
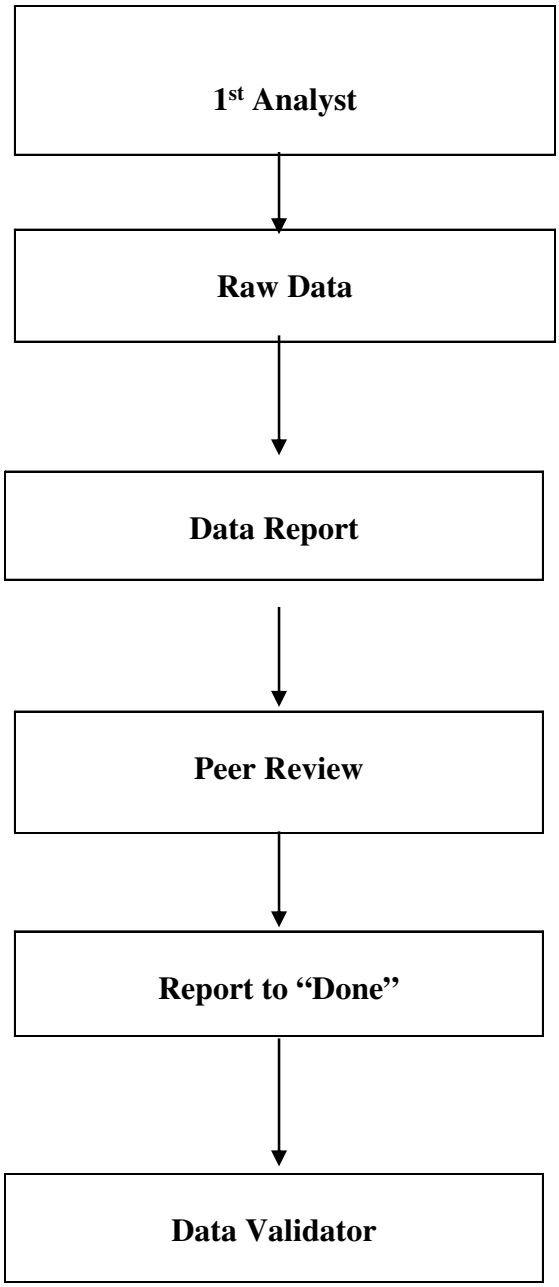


FIGURE 2: LEVELS OF REVIEW



APPENDIX 1: CALIBRATION MIXES

(FOR ILLUSTRATIVE PURPOSES ONLY)

Initial Stock Concentrations

Stock A/B:	0.5ppm	1.0ppm	2.0ppm	5.0ppm	10ppm	20ppm	50ppm	100ppm
Final Conc.								
0.2ppb	2uL							
0.5ppb	5uL							
1.0ppb		5uL						
2.0ppb			5uL					
5.0ppb				5uL				
10ppb					5uL			
20ppb						5uL		
50ppb							5uL	
80ppb								4uL
100ppb								5uL

Initial Stock Concentrations

Stock C:	5ppm	10ppm	25ppm	50ppm	100ppm	250ppm	500ppm
Stock D:	1ppm	2ppm	5ppm	10ppm	20ppm	50ppm	100ppm
Final Conc.							
5.0ppb/1ppb	5uL ea.						
10ppb/2ppb		5uL ea.					
25ppb/5ppb			5uL ea.				
50ppb/10ppb				5uL ea.			
100ppb/20ppb					5uL ea.		
250ppb/50ppb						5uL ea.	
300ppb/ **60ppb**							3uL
500ppb/100ppb							5uL ea.

APPENDIX 2: BFB MASS INTENSITY SPECIFICATIONS

Mass	Intensity Required (relative abundance)
50	15-40% of mass 95
75	30-60% of mass 95
95	base peak, 100% relative abundance
96	5-9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5-9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5-9% of mass 176

APPENDIX 3: VOLATILE ORGANIC COMPOUNDS CALIBRATION RANGES

Low level SW846 8260B and Regular level 8260B and EPA 624.1
Calibration Standard Concentration Levels

	Level 1	Level 1a	Level 2	Level 3	Level 4	Level 5	Level 6 #	Level 7 !	Level 7a
Fluorobenzene (IS)	20	20/50	20/50	20/50	20/50	20/50	20/50	20/50	20/50
1,2-Dichloroethane-d4(surr)		20/50	20/50	20/50	20/50	20/50	20/50	20/50	20/50
Dichlorodifluoromethane		0.5	1	2	5	10	20	50	100
Chloromethane		0.5	1	2	5	10	20	50	100
Vinyl chloride		0.5	1	2	5	10	20	50	100
Bromomethane		0.5	1	2	5	10	20	50	100
Chloroethane		0.5	1	2	5	10	20	50	100
Trichlorofluoromethane		0.5	1	2	5	10	20	50	100
1,1-Dichloroethene		0.5	1	2	5	10	20	50	100
Acetone	1	2.5	5	10	25	50	100	250	500
Iodomethane	1	2.5	5	10	25	50	100	250	500
Carbon disulfide	1	2.5	5	10	25	50	100	250	500
Methylene chloride		0.5	1	2	5	10	20	50	100
trans-1,2-Dichloroethene		0.5	1	2	5	10	20	50	100
1,1-Dichloroethane		0.5	1	2	5	10	20	50	100
Ethyl ether		0.5	1	2	5	10	20	50	100
Vinyl acetate	1	2.5	5	10	25	50	100	250	500
cis-1,2-Dichloroethene		0.5	1	2	5	10	20	50	100
1,2-Dichloroethene (total)		1	2	4	10	20	40	100	200
Cyclohexene		0.5	1	2	5	10	20	50	100
2-Chloroethylvinyl ether			5	10	25	50	100	250	500
2,2-Dichloropropane		0.5	1	2	5	10	20	50	100
2-Butanone	1	2.5	5	10	25	50	100	250	500
Bromochloromethane		0.5	1	2	5	10	20	50	100
Chloroform		0.5	1	2	5	10	20	50	100
1,1,1-Trichloroethane		0.5	1	2	5	10	20	50	100
1,1-Dichloropropene		0.5	1	2	5	10	20	50	100
Carbon tetrachloride		0.5	1	2	5	10	20	50	100
Benzene		0.5	1	2	5	10	20	50	100
1,2-Dichloroethane		0.5	1	2	5	10	20	50	100
Trichloroethene		0.5	1	2	5	10	20	50	100
1,2-Dichloropropane		0.5	1	2	5	10	20	50	100
Dibromomethane		0.5	1	2	5	10	20	50	100
Bromodichloromethane		0.5	1	2	5	10	20	50	100
cis-1,3-Dichloropropene		0.5	1	2	5	10	20	50	100
tert-Butylmethylether		0.5	1	2	5	10	20	50	100
Ethyl Ether			1	2	5	10	20	50	100
Acetonitrile			25	50	125	250	500	1250	2500
Methyl acetate			5	10	25	50	100	250	500
Cyclohexane			1	2	5	10	20	50	100
Methylcyclohexane			1	2	5	10	20	50	100
n-Butyl alcohol		50	100	200	500	1000	2000	5000	10000
2-Nitropropane			5	10	25	50	100	250	500
Ethyl acetate			5	10	25	50	100	250	500
Acrolein			5	10	25	50	100	250	500
Trichlorotrifluoroethane			5	10	25	50	100	250	500

APPENDIX 3: VOLATILE ORGANIC COMPOUNDS CALIBRATION RANGES cont'd

Allyl chloride			5	10	25	50	100	250	500
Acrylonitrile			5	10	25	50	100	250	500
1,4-Dioxane			50	100	250	500	1000	2500	5000
Isobutyl alcohol			50	100	250	500	1000	2500	5000
Methacrylonitrile			5	10	25	50	100	250	500
Propionitrile			5	10	25	50	100	250	500
Methyl methacrylate			5	10	25	50	100	250	500
Chlorotrifluoroethylene			5	10	25	50	100	150	200
2-Chloro-1,1,1-trifluoroethane			5	10	25	50	100	150	200
Tetrahydrofuran			5	10	25	50	100	250	500
tert-Butyl alcohol			50	100	250	500	1000	2500	5000
Isopropyl ether			1	2	5	10	20	50	100
Ethyl tert-butyl ether			1	2	5	10	20	50	100
Isopropyl alcohol			50	100	250	500	1000	2500	5000
Methyl tert-amyl ether			1	2	5	10	20	50	100
1-Chlorohexane			1	2	5	10	20	50	100
2-Chloro-1,3-butadiene(chloroprene)			1	2	5	10	20	50	100
Chlorobenzene-d5 (IS)	20	20	20/50	20/50	20/50	20/50	20/50	20/50	20/50
Toluene-d8 (surr)		20	20/50	20/50	20/50	20/50	20/50	20/50	20/50
4-Methyl-2-pentanone	1	2.5	5	10	25	50	100	250	500
Toluene		0.5	1	2	5	10	20	50	100
trans-1,3-Dichloropropene		0.5	1	2	5	10	20	50	100
1,1,2-Trichloroethane		0.5	1	2	5	10	20	50	100
Tetrachloroethene		0.5	1	2	5	10	20	50	100
1,3-Dichloropropane		0.5	1	2	5	10	20	50	100
2-Hexanone	1	2.5	5	10	25	50	20	250	500
Dibromochloromethane		0.5	1	2	5	10	20	50	100
1,2-Dibromoethane		0.5	1	2	5	10	20	50	100
Chlorobenzene		0.5	1	2	5	10	20	50	100
1,1,1,2-Tetrachloroethane		0.5	1	2	5	10	20	50	100
Ethylbenzene		0.5	1	2	5	10	20	50	100
m,p-Xylene		1	2	4	10	20	20	100	200
o-Xylene		0.5	1	2	5	10	20	50	100
Xylenes (total)		1.5	3	6	15	30	60	150	300
Stryene		0.5	1	2	5	10	20	50	100
Ethyl methacrylate			5	10	25	50	100	250	500
1,4-Dichlorobenzene-d4 (IS)	20	20	20/50	20/50	20/50	20/50	20/50	20/50	20/50
Bromofluorobenzene (surr)		20	20/50	20/50	20/50	20/50	20/50	20/50	20/50
Bromoform		0.5	1	2	5	10	20	50	100
Isopropylbenzene		0.5	1	2	5	10	20	50	100
1,1,2,2-Tetrachloroethane		0.5	1	2	5	10	20	50	100
Bromobenzene		0.5	1	2	5	10	20	50	100
1,2,3-Trichloropropane		0.5	1	2	5	10	20	50	100
n-Propylbenzene		0.5	1	2	5	10	20	50	100
2-Chlorotoluene		0.5	1	2	5	10	20	50	100
1,3,5-Trimethylbenzene		0.5	1	2	5	10	20	50	100
4-Chlorotoluene		0.5	1	2	5	10	20	50	100

APPENDIX 3: VOLATILE ORGANIC COMPOUNDS CALIBRATION RANGES CONT'D

1,2,4-Trimethylbenzene		0.5	1	2	5	10	20	50	100
sec-Butylbenzene		0.5	1	2	5	10	20	50	100
1,3-Dichlorobenzene		0.5	1	2	5	10	20	50	100
tert-Butylbenzene		0.5	1	2	5	10	20	50	100
Isopropyltoluene		0.5	1	2	5	10	20	50	100
1,4-Dichlorobenzene		0.5	1	2	5	10	20	50	100
n-Butylbenzene		0.5	1	2	5	10	20	50	100
1,2-Dichlorobenzene		0.5	1	2	5	10	20	50	100
1,2-Dibromo-3-chloropropane		0.5	1	2	5	10	20	50	100
1,2,4-Trichlorobenzene		0.5	1	2	5	10	20	50	100
Hexachlorobutadiene		0.5	1	2	5	10	20	50	100
Naphthalene		0.5	1	2	5	10	20	50	100
1,2,3-Trichlorobenzene		0.5	1	2	5	10	20	50	100
cis-1,4-Dichloro-2-butene			5	10	25	50	100	250	500
trans-1,4-Dichloro-2-butene			5	10	25	50	100	250	500
Pentachloroethane			5	10	25	50	100	250	500
Benzyl chloride			5	10	25	50	100	250	500
Cyclohexanone			25	50	125	250	500	1250	2500
bis(2-Chloro-isopropyl)ether			5	10	25	50	100	250	500

Method	PQL	Concentration range		
SW 846 8260B low level	Level 1&1a	Levels 1-> 7a	IS/SS @ 20 ppb	
SW846 8260B/624.1	Level 2	Levels 1a-> 7a	IS/SS @ 50 ppb	n-butyl alcohol only in 1a

#: Indicates calibration verification concentration level used for low level analysis

!: Indicates calibration verification concentration level used for regular level analysis

APPENDIX 4: Method 8260C Criteria

In addition to the general criteria outlined in the body of this SOP, the following requirements must be met prior to analysis of samples requesting 8260C.

1.0 Initial Calibration

1.1 The calibration curve must be prepared from a minimum of five calibration points. The RSD for all compounds must be less than 20%. If the RSD of any target analyte exceeds 20%, then use one of the options in section 15.2.13.

1.2 If more than 10% of the compounds included in the initial calibration exceed the 20% RSD and do not meet the minimum correlation coefficient of 0.990 for linear regression, system maintenance should be performed and a new calibration curve must be analyzed.

1.3 For compounds utilizing the method of linear regression, a minimum quantitation check should be performed on the lowest calibration point. This involves requantitation of the lowest points of the ICAL as samples (not as calibration levels) and evaluating the recalculated concentrations compared to the true concentrations present. The recalculated concentrations of the low calibration standards should be within $\pm$ 30% of the standard's true concentration. It should be noted that not all compounds are present in the calibration levels at the same concentrations. Documentation is made on the ICAL Validation Check Sheet employed by the data validators.

1.4 It is also recommended that a minimum response factor for the most common target analytes be demonstrated for each individual calibration level. See Appendix 5 for a list of compounds and recommended responses. Due to the large number of compounds analyzed by this method, some compounds may fail to meet the criteria. These compounds may not be critical to the specific project and may be used as qualified data or as estimated values.

1.5 SPCC and CCC compounds are not recognized in method 8260C.

2.0 Initial Calibration Verification

2.1 Calibration curves must be verified using an initial calibration verification standard. This must be a second source standard from the initial calibration. It may be from a different vendor or may be a different lot from the same vendor. The response factor or true value (percent difference or drift) should not exceed $\pm$ 30%. Documentation is made on the ICAL Validation Check Sheet employed by the data validators.

2.2 In the event that a compound falls outside of the $\pm$ 30% acceptance criteria, possible courses of action include reanalysis of the ICV, analysis of a different ICV standard (lot or vendor), maintenance to the analytical system and/or recalibration. If holding times are expiring, sample analysis may proceed. In this case, the Project Manager is notified, documentation is made in the case narrative and the data are qualified.

APPENDIX 4: METHOD 8260C CRITERIA CONTINUED

3.0 Continuing Calibration Verification Analysis

3.1 Each of the most common target analytes in the calibration verification standard should meet the minimum response factors in Appendix 5. If the minimum response factors are not met, check for standard degradation, injection port contamination, column contamination, active sites, or moisture control loss.

3.2 The percent drift or percent difference for each analyte of interest should be +/-20%. The calibration verification standard may be from the same source as the midpoint of the initial calibration or may be a different source. If more than 20% of the total number of requested compounds exceed +/-20% requirement, corrective action should be taken and no samples analyzed. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when a failed compound is present, the concentrations must be reported as estimated values.

4.0 Internal Standard Retention Time Monitoring

The retention times of the internal standards in the calibration verification standard must be evaluated. If the retention time for any internal standard changes by more than 10 seconds from that in the mid-point standard level of the initial calibration, the system must be investigated for malfunctions and reanalysis of all samples analyzed during this period is required.

5.0 Surrogate Monitoring

The recommended surrogates for this method are Toluene-d8, 4-Bromofluorobenzene, and 1,2-Dichloroethane-d4.

APPENDIX 5: RECOMMENDED RESPONSE FACTORS FOR METHOD 8260C

TABLE 4

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382

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APPENDIX 5: RECOMMENDED RESPONSE FACTORS FOR 8260C CONT'D

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

[a] The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.
[b] Data provided by EPA Region III laboratory.

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APPENDIX 6: POOR PURGING COMPOUNDS

Dichlorodifluoromethane
Trichlorofluoromethane
Trichlorotrifluoroethane
Acetone
2-Butanone
2-Hexanone
4-Methyl-2-pentanone
Cyclohexanone
Carbon disulfide
n-Butyl alcohol
tert-Butyl alcohol
Isobutyl alcohol
Isopropyl alcohol
Vinyl acetate
Methyl acetate
Ethyl acetate
1,4-Dioxane
Methyl methacrylate
Ethyl methacrylate
Propionitrile
Methacrylonitrile
1,2-Dibromo-3-chloropropane

APPENDIX 7: TENTATIVE IDENTIFICATION PROCEDURES

1. Relative intensities of major ions (> 10%) in the reference spectrum should be present in sample spectrum. This equates to detects of TICs $\geq$ 5 ppb concentration (10% of 50 ppb internal standard concentration).

2. Relative intensities of the major ions should agree within $\pm$ 30% (i.e., for an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 20 and 80 percent.)

3. Molecular ion present in reference spectrum should be present in sample spectrum.

4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

5. If no valid identification can be made, the compound should be reported as "unknown." If an additional classification can be given to the unknown (unknown hydrocarbon, aromatic, chlorinated, etc.) it should be listed as such.

6. Peaks that are detected in the sample and analytical blank should be flagged as such on the report.

7. TIC detects with “fit” value < 85 will be identified with J qualifier. Detects with “fit” value > 85 will be identified with NJ qualifier and parameter name.

8. The CAS for calibrated compounds not included on the client requested list of parameters will be the true CASRN, while the CASRN for non-calibrated compounds will be reported with leading zeros (000) in front of the probable CASRN.

9. GEL’s policy for reporting calibrated compounds when requested by the client as TICs only is to follow TIC reporting from National Functional Guidelines and report only $\geq$ 5ppb for VOA with J or NJ qualification.

APPENDIX 8: STANDARD METHOD 6200

1. Perform continuing calibration with one or more of the concentrations of analytical standards in the initial calibration. Vary actual concentration of continuing calibration standard over calibration range, with a minimum concentration greater than 2 times the reporting limit, the acceptance criteria is 70%- 130%. For gases, the acceptance criteria is 60%-140%. If criteria is not met then re-analyze continuing calibration standard or re-analyze initial calibration.

2. The Continuing Calibration Standard must be the same source (lot number) as the Initial Calibration Standard.

3. Internal Standard responses should be in the range ± 30% compared to the mean calibration curve area response.

4. The LCS/LFB/MS/MSD/LFS are from a different primary mix than that used to develop the initial calibration.

5. A closing continuing calibration standard (same lot number) must be analyzed. The acceptance criteria is 70%-130%. For Gases the acceptance criteria is 60%-140%. The concentration of this standard is varied and not the same as the continuing calibration standard.

APPENDIX 9: 4-BROMOFLUOROBENZENE (BFB) SUGGESTED CRITERIA FOR 8260D

m/z	Intensity (relative abundance)
95	50-200% of mass 174
96	5 to 9% of m/z 95 (5 to 15% when using H_2 carrier)
173	<2% of *m/z* 174
174	50-200% of mass 95
175	5 to 9% of *m/z* 174
176	95 to 105% of *m/z* 174
177	5 to 10% of *m/z* 176

*Criteria based on EPA Method 524.4 (Reference 17), with modified m/z and m/z 174 abundance criteria.

APPENDIX 10: SUMMARY OF QC CRITERIA FOR USE WITH 8260D

SUMMARY OF QC CRITERIA FOR USE WITH 8260D[a]

Quality Control Type	Minimum frequency	Specification	Suggested Acceptance Criteria
Instrument performance check (Secs. 9.3.1, 11.3.1)	Prior to initial calibration	Must be verified prior to initial calibration	Meet ion ratio criteria for reference compound: 4-Bromofluorobenzene Appendix 9, or alternative documented criteria
Initial Calibration (ICAL) (Secs. 9.3.2, 11.3.2-11.3.5)	Prior to analyzing samples, and as needed if continuing performance criteria cannot be met	5 points minimum for RF and linear regressions, 6 points minimum for quadratic regressions; >90% of reported target analytes meet initial calibration criteria	For average response factor (RF) calibration model: ≤20% RSD of RFs; For linear or quadratic regression model: R≥0.995, R^2≥0.99; Independent of calibration model: LLOQ standard recalculation (refit) is within ±50% of true value if it is the low calibration point; All other standards within ±30% of true value; Or, relative standard error (RSE) ≤20% (Refer to Method 8000 and Reference 16 for calculation) See Method 8000 for additional criteria.
ICAL Verification (ICV) (Secs. 9.3.2, 11.3.6)	After each initial calibration, and prior to analyzing samples	Prepared from different source of target analytes than initial calibration standards	Calculated concentrations of target analytes are within ±30% of true value
Continuing Calibration Verification (CCV) (Secs. 9.3.3, 11.4)	Once every 12 hours	>80% of target analytes meet CCV criteria	Targets are ≤20% difference or drift; IS responses are within 50% to 200% of mid-point of ICAL or average of ICAL ISs; and RTs for ISs have not shifted >30 seconds relative to ICAL
Blanks (Secs. 9.5, 9.6.1)	One method blank per preparation batch of 20 or fewer samples; other blanks as needed	NA	Target analyte concentrations in blanks are <1/2 LLOQ, or ≤ 10% of concentration in field samples
Laboratory Control Sample (LCS) (Sec 9.6.2)	One per preparation batch of 20 or fewer samples	NA	Meets recovery criteria (CCV criteria may be used if LCS and CCV are identical)

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APPENDIX 10: SUMMARY OF QC CRITERIA FOR THE USE WITH 8260D (CONTINUED)

Quality Control Type	Minimum frequency	Specification	Suggested Acceptance Criteria
Duplicates and Matrix Spikes (Secs. 9.6.3)	A duplicate and matrix spike, or matrix spike/matrix spike duplicate per preparation batch of 20 or fewer samples, provided adequate material is made available to the laboratory	NA	Meets performance-based or project-defined recovery criteria for matrix spikes; Meets relative % difference between measured concentrations in sample and laboratory duplicate or in matrix spike/matrix spike duplicate;
Surrogates (Secs. 9.7)	Added to each sample	NA	Meets performance-based recovery criteria established by the laboratory or criteria chosen for the project
Internal Standards (Secs. 9.8, 11.5.6)	Added to each sample	NA	IS response is within 50 - 200% of the response of the same IS in the midpoint ICAL standard (or average of ICAL) or most recent CCV
Qualitative Analyte Identification (Sec. 11.6.1)	Each target analyte	NA	RT in sample is within ±10 sec of RT in midpoint ICAL or CCV standard or within ±10 seconds relative to the shift of the associated IS (delta RT of the IS ± 10 seconds) Characteristic ion(s) are within ±30% of expected ion ratio in reference spectrum; or, match to reference library spectra ≥0.8 (only for full mass range acquisition modes)

[a] Default acceptance criteria; alternative criteria may be specified for a given application. Refer to Sec. 9 for more information.

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